Claims

What is claimed is:

2. A sequential processing reaction vessel for treatment of solids at high temperatures and pressures comprising:

a pressure resistant, microwave transparent, outer housing, said housing being able to withstand at least 150 psi of internal pressure at temperatures up to at least 150° C;

a chemically inert, microwave transparent, inner housing, said housing positioned within a cavity formed by said outer housing, said inner housing being resistant to reaction with corrosive liquids at temperatures up to at least 150° C and internal pressures up to at least 150 psi;

a chemically inert, microwave transparent, filter member, said filter positioned within said inner housing in a substantially horizontal orientation to accommodate placement and retention of a solid sample material, said filter having a pore size which is smaller than a typical particle size of said solid material, said filter permitting retention of said solid material and passage of said corrosive liquids, said filter being resistant to corrosive liquids at temperatures up to at least 150° C and pressures up to at least 150 psi;

a chemically inert, microwave transparent, top valve, said top valve having an open position for permitting introduction of said corrosive liquids to a reactor volume formed by said inner housing; said top valve having a closed position which provides a leak-proof seal of said vessel at temperatures up to at least 150° C and pressures up to at least 150 psi; and

a chemically inert, microwave transparent, bottom valve, said bottom valve having an open position for permitting removal of said filtered corrosive liquids from said vessel after reactive contact with said solid sample, said bottom valve having a closed position which provides a leak-proof seal of said vessel at temperatures up to at least 150° C and pressures up to at least 150 psi.

3. The vessel of claim 2 further comprising a chemically inert, microwave transparent filter frit positioned within said inner housing below said membrane filter in a substantially

horizontal orientation, said frit having a pore size which is substantially larger than said membrane filter pore size, said frit supporting said filter and permitting passage of said corrosive liquids, said frit being resistant to corrosive liquids at temperatures up to at least 150° C and pressures up to at least 150 psi;

4. The vessel of claim 2 wherein said filter member comprises a laminated filter assembly comprising a porous, polytetrafluoroethylene membrane filter top layer having a predominately sub-micron pore size, a porous, polytetrafluorethylene filter frit bottom layer having a pore size which is substantially larger than the pore size of said membrane filter, and a non-porous, polytetrafluoroethylene toroidal outer ring for engaging an o-ring seal.

5. The vessel of claim 2 wherein said top and bottom valves further comprise a pressure relief seal which ruptures at excessive vessel pressures.

6. The vessel of claim 2 further comprising auxiliary ports in said top cover to accommodate a temperature sensor and pressure sensor for monitoring vessel reaction conditions.

7. The vessel of claim 2 wherein said outer housing is comprised of a material selected from the group consisting of polyetherimide, Ultem® or Ultem 2300®, said inner housing is comprised of a material selected from the group consisting of PTFE, PFA or Teflon®, said filter is comprised of a material selected from the group consisting of treated hydrophobic polytetrafluoroethylene or hydrophilic polytetrafluoroethylene, and said top and bottom valves are comprised of a material selected from the group consisting of PTFE, PFA or Teflon®.

8. A sequential processing reaction vessel for treatment of solids at high temperatures and pressures comprising:

a chemically inert, microwave transparent, outer housing comprised of

a threaded top collar member secured to a top threaded end of a hollow cylindrical exterior jacket, said top collar having a concentric center opening for insertion of an elongated cylindrical top member of a top cover of an inner housing, and

a threaded bottom ring member secured to a bottom threaded end of said exterior

jacket, said bottom ring having a concentric center opening for insertion of an elongated cylindrical bottom member of a filter holder of said inner housing;

a chemically inert, microwave transparent, said inner housing comprised of

said inner housing top cover urged against a top cover o-ring and an interior liner by said top collar member secured to said top end of said exterior jacket, said top cover having a concentric bore extending through said elongated top member from an exterior top end to an interior bottom surface of said top cover, said elongated member top end having a threaded exterior surface, said top cover having a concentric outer shoulder on a top surface for engaging said top collar, said top cover having a beveled outside edge on said cover bottom surface for engaging said top cover o-ring;

a cylindrical interior liner having an outside cylindrical surface mating with an interior cylindrical surface of said exterior jacket, said interior liner top end having an inner shoulder for receiving and engaging said top cover o-ring, said interior liner bottom end having an outer shoulder for engaging said filter holder, said liner outer shoulder having a beveled outside bottom edge,

a filter holder o-ring engaged by said beveled edge of said interior liner bottom shoulder, said filter holder urged against said filter holder o-ring and said interior liner by said threaded bottom ring member secured to said bottom end of said exterior jacket, said filter holder top surface having a primary cylindrical cavity for engaging said liner bottom end shoulder, said primary cavity having a bottom surface for supporting said filter and an inside bottom edge for receiving and

engaging said filter holder o-ring, said primary cavity bottom surface having a shallow secondary cylindrical cavity for collecting filtered liquids, said secondary cavity bottom surface having a cylindrical bore extending through said elongated bottom member to an externally threaded distal end, said filter holder bottom surface having a concentric external shoulder for engaging said threaded bottom ring;

a chemically inert, microwave transparent, filter member supported by said primary cavity bottom surface, said filter urged against said primary cavity bottom surface, said filter holder o-ring and said inner liner by said threaded bottom ring member secured to said jacket bottom end;

a chemically inert, microwave transparent threaded top valve, said top valve threads engaged with said threaded distal end of said top elongated member, said top valve having an open position for permitting introduction of corrosive liquids to a reactor volume formed by said inner housing; said top valve having a closed position for sealing said vessel at temperatures up to at least 150° C and pressures up to at least 150 psi; and

a chemically inert, microwave transparent, threaded bottom valve, said bottom valve threads engaged with said threaded distal end of said bottom elongated member, said bottom valve having an open position for permitting removal of filtered corrosive liquids from said reactor volume after reactive contact with said solid sample, said bottom valve having a closed position for sealing said vessel at temperatures up to at least 150° C and pressures up to at least 150 psi.

9. The vessel of claim 8 wherein said filter member comprises a laminated filter assembly comprising a porous, polytetrafluoroethylene membrane filter top layer having a predominately sub-micron pore size, a porous, polytetrafluorethylene filter frit bottom layer having a pore size which is substantially larger than the pore size of said membrane filter, and a non-porous, polytetrafluoroethylene outer ring for engaging said filter holder o-ring.

10. The vessel of claim 8 further comprising a chemically inert, microwave transparent, filter frit positioned below said filter member and supported by said secondary cavity bottom surface.

11. The vessel of claim 8 wherein said top and bottom valves further comprise a pressure relief seal which ruptures at excessive vessel pressures.

12. The vessel of claim 8 further comprising at least one pair of alignment pins and associated alignment pin bores for alignment and assembly of said interior liner and said filter holder.

13. The vessel of claim 8 further comprising auxiliary ports in said top cover to accommodate a temperature sensor and pressure sensor for monitoring vessel reaction conditions.

14. The vessel of claim 8 wherein said inside surface of said interior liner has a tapered cross section which narrows at the liner bottom.

15. The vessel of claim 8 wherein said outer housing is comprised of a material selected from the group consisting of polyetherimide, Ultem® or Ultem 2300®, said inner housing is comprised of a material selected from the group consisting of PTFE, PFA or Teflon®, said filter is comprised of a material selected from the group consisting of treated hydrophobic polytetrafluoroethylene or hydrophilic polytetrafluoroethylene, and said top and bottom valves are comprised of a material selected from the group consisting of PTFE, PFA or Teflon®.

16. A method for sequential processing and reaction of solids at high temperatures and pressures comprising the steps of:

providing a sequential processing reaction vessel comprised of

a pressure resistant, microwave transparent, outer housing, said housing being able to withstand at least 150 psi of internal pressure at temperatures up to at least 150EC;

a chemically inert, microwave transparent, inner housing, said housing positioned within a cavity formed by said outer housing, said inner housing being resistant to reaction with corrosive liquids at temperatures up to at least 150° C and internal pressures up to at least 150 psi;

a chemically inert, microwave transparent, filter member, said filter positioned within said inner housing in a substantially horizontal orientation to accommodate placement and retention of a solid sample material, said filter having a pore size which is smaller than a typical particle size of said solid material, said filter permitting retention of said solid material and passage of said corrosive liquids, said filter being resistant to corrosive liquids at temperatures up to at least 150° C and pressures up to at least 150 psi;

a chemically inert, microwave transparent, top valve, said top valve having an open position for permitting introduction of said corrosive liquids to a reactor volume formed by said inner housing; said top valve having a closed position which provides a leak-proof seal of said vessel at temperatures up to at least 150° C and pressures up to at least 150 psi; and

a chemically inert, microwave transparent, bottom valve, said bottom valve having an open position for permitting removal of said filtered corrosive liquids from said vessel after reactive contact with said solid sample, said bottom valve having a closed position which provides a leak-proof seal of said vessel at temperatures up to at least 150° C and pressures up to at least 150 psi;

placing a weighed sample on a top surface of said filter;

sealing said inner housing and said bottom valve;

introducing an initial treatment solution through an opening in said top valve;

closing said top valve;

monitoring an internal temperature and pressure of said vessel;

heating said vessel in a microwave oven according to a predetermined temperature and pressure cycle;

cooling said vessel to room temperature;

collecting said initial treatment solution containing extracted analytes from said bottom valve by opening said top valve and said bottom valve;

rinsing said vessel with a solvent to collect residual initial treatment solution and analytes;

closing said bottom valve;

introducing at least one additional treatment solution through said opening in said top valve; and

repeating said introducing, monitoring, heating, cooling, collecting, opening, rinsing and closing steps with said at least one additional treatment solution.

17. The method of claim 16 further comprising the step of analyzing the extracted analytes in each of said treatment solutions.

18. The method of claim 16 wherein said collecting step comprises applying vacuum to said bottom valve.

19. The method of claim 16 wherein said collecting step comprises applying pressure to said top valve.

20. The method of claim 16 wherein at least five different treatment solutions are employed.

21. The method of claim 16 wherein at least five different temperature and pressure cycles are employed.

22. A multiple sequential processing reaction vessel system for treatment of solids at high temperatures and pressures comprising:

a plurality of sequential processing reaction vessels comprised of

a pressure resistant, microwave transparent, outer housing, said housing being able to withstand at least 150 psi of internal pressure at temperatures up to at least 150° C;

a chemically inert, microwave transparent, inner housing, said housing positioned within a cavity formed by said outer housing, said inner housing being resistant to reaction with corrosive liquids at temperatures up to at least 150° C and internal pressures up to at least 150 psi;

a chemically inert, microwave transparent, filter member, said filter positioned within said inner housing in a substantially horizontal orientation to accommodate placement and retention of a solid sample material, said filter having a pore size which is smaller than a typical particle size of said solid material, said filter permitting retention of said solid material and passage of said corrosive liquids, said filter being resistant to corrosive liquids at temperatures up to at least 150° C and pressures up to at least 150 psi;

a chemically inert, microwave transparent, top valve, said top valve having an open position for permitting introduction of said corrosive liquids to a reactor volume formed by said inner housing; said top valve having a closed position for sealing said reactor volume at temperatures up to at least 150° C and pressures up to at least 150 psi; and

a chemically inert, microwave transparent, bottom valve, said bottom valve having an open position for permitting removal of said filtered corrosive liquids from said vessel after reactive contact with said solid sample, said bottom valve having a closed position for sealing said reactor volume at temperatures up to at least 150° C and pressures up to at least 150 psi; and

a carousel assembly for supporting said vessels comprised of

a support pedestal;

a bottom plate attached to said support pedestal in a substantially horizontal configuration, said bottom plate configured with a plurality of openings to receive said bottom valve assembly of said vessels and provide access to said bottom valves;

a top plate attached to said support pedestal above said bottom plate in a substantially horizontal configuration, said top plate configured with a plurality of

openings to receive said outer housing of said vessels and provide access to said top valves and said vessels;

wherein each of said plurality of outer housing openings in said top plate is vertically aligned and paired with one of said bottom valve assembly openings in said bottom plate to support said plurality of vessels in said carousel assembly.

23. The system of claim 22 further comprising at least one temperature sensor and pressure sensor for monitoring reactions in at least one of said vessels.

24. The system of claim 22 further comprising a rotating pedestal for mounting said carousel assembly with said vessels, said rotating pedestal providing for rotation of said carousel assembly in either a clockwise or counter-clockwise direction to achieve uniform heating of said plurality of vessels when said carousel assembly is placed in an oven.

SEQUENTIAL PROCESSING REACTION VESSEL FOR CHEMICAL FRACTIONATION AND ANALYSIS

A sequential processing reactor vessel and method is disclosed for accelerated extraction and fractionation of chemical analytes from complex solid sample materials. The device and method enable sequential extraction of elemental constituents from solid materials by sequentially contacting target samples within a single reaction vessel using a series of different reagents at temperatures as high as 150° C and pressures up to 150 psi to accelerate reactions. The aggressive chemical treatments provided by the disclosed device and method provide for complete digestion of solid samples in liquid analyte samples that can be directly analyzed by conventional spectrometry or other suitable methods. The disclosed device and method provide for efficient sample processing and accelerated reactions to significantly reduce processing times and costs for elemental analysis of solids while improving accuracy, precision and reliability of results compared to conventional devices and methods. The disclosed device and method are compatible with both conventional convection and radiant heating sources as well as microwave heating and can be readily adapted to marine, geological, environmental, industrial and research solids analysis applications.